

PATENT SPECIFICATION

NO DRAWINGS

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1,196,595



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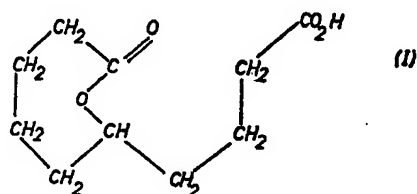
COMPLETE SPECIFICATION

Preparation of a Lactone

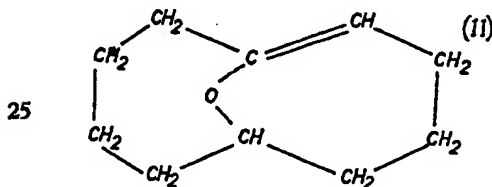
We GEIGY (U.K.) LIMITED, a British Company, whose Registered Office address is Simonsway, Manchester 22, Lancashire, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The present invention relates to a novel chemical compound and a process for the preparation thereof and in particular to a novel lactone and a process for the production thereof.

According to the present invention, there is provided the novel compounds, 6 - hydroxy - 1,10 - decanedioic acid - 1,6 - lactone having the formula I:—



According to a second aspect of the present invention, there is provided a process of producing 6 - hydroxy - 1,10 - decanedioic acid - 1,6 - lactone comprising treating 1,6-oxido - cyclodec - 1 - ene having the formula:—



[Price 5s. 0d. (25p)]

with ozone and thermally oxidising the ozonised material at an elevated temperature.

The initial ozonisation procedure is conveniently conducted in the presence of a solvent consisting of an aliphatic alcohol or a monocarboxylic acid each being of straight-chain and having from one to four carbon atoms in the carbon chain. Suitable alcohols include, for instance, methanol and ethanol. If a monocarboxylic acid is used, it may be, for instance, acetic acid or propionic acid. The 1,6 - oxidocyclodec - 1 - ene used as reactant is advantageously dissolved in the alcohol or acid solvent before being contacted with ozone.

The ozonisation is conveniently effected using ozone in the form of ozonised oxygen. The proportion of ozone in the ozonised oxygen stream may vary but is preferably within the range of from 1% to 5% by weight.

In order to minimise volatilisation of the reactant and/or the ozonisation solvent, the ozonisation procedure is preferably effected at a temperature below 0°C. The ozonisation is desirably carried out until all or substantially all of the ethylenically unsaturated double bond in the compound of formula II has been converted to the corresponding ozonide. The completion of the ozonisation process may be detected, for example, by passing the effluent gases from the ozonisation reaction mixture through an aqueous solution of potassium iodide and boric acid; the precipitation of free iodine marking the completion of the ozonisation reaction.

The ozonide so produced is preferably partially purified by distilling therefrom any ozonisation solvent before the ozonide is subjected to thermal oxidation.

The oxidation decomposition of the ozonide, produced in the first stage of the reaction, is preferably effected at an elevated temperature by means of hydrogen peroxide in a fatty

acid solution of the ozonide, for example, a formic acid solution of the ozonide. Alternatively, the oxidative decomposition of the ozonide may be effected by means of a stream of gaseous oxygen or ozonised oxygen, the remaining conditions being otherwise the same.

The oxidative decomposition of the ozonide may be effected for instance at temperatures up to the boiling point of the reaction mixture. However, the oxidative decomposition of the ozonide using hydrogen peroxide as the oxidising agent is normally and conveniently carried out by gently heating the reaction mixture until vigorous exothermic reaction sets in. After the exothermic reaction has subsided, heating of the reaction mixture under reflux conditions for a short period of time, for instance, up to one hour is usually sufficient to complete the oxidative decomposition.

The 6 - hydroxy - 1,10 - decanedioic acid - 1,6 - lactone so produced may be purified by removal of any solvent employed during the oxidative decomposition process. In this way, the lactone product may be isolated as an oil and in a form very suitable for conversion to 5-hydroxysebacic acid by means of the alkaline hydrolysis process described and claimed in our co-pending British patent application No. 21513/68 (Serial No. 1196596), which, in turn, may be converted into sebacic acid according to the process described and claimed in the parent application, British patent application No. 52602/66 (Serial No. 1196594).

The present invention is further illustrated by the following Example. Parts by weight expressed therein bear the same relation to parts by volume as do kilograms to litres.

EXAMPLE

1,6 - Oxidocyclodec - 1 - ene may be produced, for example, from 1 - acetoxy - 1,6 - oxidocyclodecane as described in Example 1 of our co-pending British patent application No. 21514/68 (Serial No. 1196597) or it may also be obtained according to Example 3 of said application.

15.2 parts of 1,6 - oxidocyclodec - 1 - ene dissolved in 100 parts by volume of methanol was treated at $-40^{\circ}\text{C}.$, with a stream of ozonised oxygen until the effluent gases gave a ready precipitate of iodine from an aqueous potassium iodide/boric acid solution. The methanol solvent was removed at $25^{\circ}\text{C}.$, under reduced pressure, and to the remaining oil there was added 70 parts by volume of 90% weight/weight formic acid and 20 parts by volume of 50% weight/weight hydrogen peroxide. This mixture was heated gently until a vigorous exothermic reaction set in. After the initial reaction had subsided, external heating at $100^{\circ}\text{C}.$, was continued for 15 minutes and the mixture was finally heated

under reflux conditions for a further 30 minutes.

The water and formic acid were removed from the reaction mixture under reduced pressure. The oil remaining was then distilled under high vacuum and provided the following fractions:—

(i) 2.7 parts of a liquid having a boiling range of up to $212^{\circ}\text{C}.$ at 0.2 millimetres of mercury pressure,

(ii) 13.1 parts of a liquid having boiling point of $212^{\circ}\text{C}.$ at 0.2 millimetres of mercury pressure, and

(iii) 4.5 parts of a high-boiling residue.

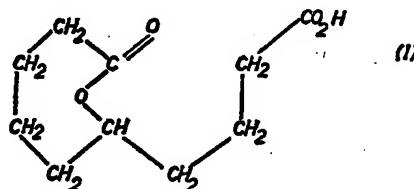
The main fraction boiling at $212^{\circ}\text{C}.$ (0.2 millimetres) was partially crystalline in form and had an acid value of 309 milligrams of KOH per gram (6 - hydroxy - 1,10 - decanedioic acid 1,6-lactone has a theoretical acid value of 280 milligrams of KOH per gram).

Re-crystallisation of the crude partially-crystalline product from diethyl ether at $0^{\circ}\text{C}.$ gave white prisms having melting-point of $46^{\circ}\text{C}.$, an acid value of 281 milligrams of KOH per gram and the following elemental analysis by weight:—

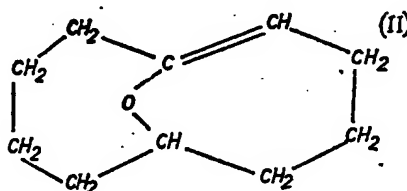
	Found	Required (for $\text{C}_{10}\text{H}_{16}\text{O}_4$)
carbon	59.79	59.98
hydrogen	7.63	8.05

WHAT WE CLAIM IS:—

1. The compound, 6 - hydroxy - 1,10 - decanedioic acid-1,6-lactone, having the formula:—



2. A process of producing 6 - hydroxy - 1,10 - decanedioic acid-1,6-lactone comprising treating 1,6 - oxido - cyclodec - 1 - ene having the formula:—



- with ozone, and thermally oxidising the ozonised material at an elevated temperature.
3. A process as claimed in claim 2 wherein the ozonisation is conducted in the presence of a solvent consisting of an aliphatic alcohol or a monocarboxylic acid each being of straight-chain and having from 1 to 4 carbon atoms in the chain.
4. A process as claimed in claim 3 wherein the alcohol is methanol or ethanol.
5. A process as claimed in claim 3 wherein the acid is acetic acid or propionic acid.
6. A process as claimed in any of claims 3 to 5 wherein the 1,6 - oxidocyclodec - 1 - ene is dissolved in the alcohol or acid solvent before being contacted with ozone.
7. A process as claimed in any of claims 2 to 6 wherein the ozone is in the form of ozonised oxygen.
8. A process as claimed in claim 7 wherein the proportion of ozone in the ozonised oxygen is within the ranges of from 1% to 5% by weight.
9. A process as claimed in any of claims 2 to 8 wherein the ozonisation is effected at a temperature below 0°C.
10. A process as claimed in any of claims 2 to 9 wherein the ozonisation is carried out until all or substantially all of the ethylenically unsaturated double bond in the starting-material has been converted to the corresponding ozonide.
11. A process as claimed in any of claims 2 to 10 wherein the ozonide produced is partially purified by distilling therefrom any ozonisation solvent before subjecting the ozonide to thermal oxidation.
12. A process as claimed in any of claims 2 to 11 wherein the oxidative decomposition of the ozonide is effected at an elevated temperature by means of hydrogen peroxide in a fatty acid solution of the ozonide.
13. A process as claimed in any of claims 2 to 11 wherein the oxidative decomposition of the ozonide is effected at an elevated temperature by means of gaseous oxygen or ozonised oxygen in a fatty acid solution of the ozonide.
14. A process as claimed in claim 12 or 13 wherein the fatty acid solvent is formic acid.
15. A process as claimed in any of claims 2 to 14 wherein the oxidative decomposition of the ozonide is effected at a temperature up to the boiling point of the reaction mixtures.
16. A process as claimed in any of claims 2 to 12 or 14 or 15 wherein the oxidative decomposition of the ozonide using hydrogen peroxide is effected by gently heating the reaction mixture until a vigorous exothermic reaction sets in and, after the exothermic reaction has subsided, heating the reaction mixture under reflux conditions for a period of up to one hour.
17. A process as claimed in any of claims 2 to 16 wherein the 6 - hydroxy - 1,10 - decanedioic acid-1,6-lactone is purified by removal of any solvent employed during the oxidative decomposition process.
18. A process of producing 6 - hydroxy - 1,10 - decanedioic acid-1,6-lactone substantially as described in the Example.
19. 6 - Hydroxy - 1,10 - decanedioic acid-1,6-lactone when produced by a process as claimed in any of claims 2 to 18.
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